Ultrafast self-catalytic growth of silicon carbide nanowires

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Silicon carbide nanowires (SiCNWs) (with diameters of tens of nanometer and aspect ratio well above 10^3), consisting of β -SiC one-dimensional single crystals wrapped in amorphous nitrogencontaining SiO₂ sheaths, were efficiently synthesized in gram quantities by autogenous combustion synthesis using Si as a defluorination reagent of poly(tetrafluoroethylene). The combustion temperature was evaluated using the emission spectroscopy. Vapor–liquid–solid mechanism of a self-catalytic growth of the SiCNWs is proposed.

I. INTRODUCTION

The synthesis of one-dimensional (1D) nanostructures has recently attracted tremendous interest due to their unique properties.¹ Specifically, nanowires (NWs) often show distinct properties from their bulk counterparts because of radial confinement² that makes them promising for numerous applications, mostly in electronics and materials science. Silicon carbide possesses excellent chemical, thermal, mechanical, and electronic properties,³ and SiC nanowires (SiCNWs) can be used as interconnects in integrated circuits and they are also compatible with the processing of electronic devices. However, all synthesis techniques of SiCNWs involve complicated processes and usually long-duration reactions.

To overcome those drawbacks, we describe a simple, easy to operate, and one-step efficient SiCNWs production via transformation of poly(tetrafluoroethylene) (PTFE) into SiCNWs using elemental Si during selfpropagating high-temperature synthesis (SHS).⁴ The very high temperatures/pressures and rapid movement of the reaction front wave, with its resultant local self-quenching, can yield unique micro- and nanostructured compositions.⁵ PTFE has already been shown to be effectively defluorinated using different reducers by fast combustion synthesis.⁶ The use of Si-bearing reducers resulted in the efficient formation of 1D SiC.⁷ The molar ratio of powdered reactants was balanced to produce just salt and soot as byproducts as given in the equation:

$$2nSi + (CF_2CF_2)_n \rightarrow nSiC + nC + nSiF_4$$

The transformation mechanism from starting reactants to SiCNWs has not been clarified yet, and the present study of the growth process and characterization of nanostructures is obviously important. Various SiC growth conditions can affect the reaction yield. These variables include (but are not limited to): growth technique, starting reactant mixture characterization (mass, reducer/oxidizer ratio), gas pressure, and combustion atmosphere. The last one seems to be crucial since the synthesis evidently proceeds via gas phase intermediates, i.e., one can see a significant qualitative difference between Si/PTFE mixtures combusted in air and in argon.

II. EXPERIMENTAL

A. Materials

PTFE (particle size below 1 μ m; Aldrich, Steinheim, Germany) and elemental Si (particle size below 43 μ m, 99%; Aldrich, Steinheim, Germany) were thoroughly mixed and used in the proper stoichiometric proportion.

B. Methodology for the combustion synthesis

Typically, combustion synthesis was carried out in a stainless steel reaction vessel modeled after a bomb calorimeter under 10 atm initial pressure at either argon or air atmosphere. The high-pressure reactor was provided with an observation port for emission spectroscopy diagnostics since the light emitted during SiC synthesis was used to estimate the reaction temperature. The fast combustion of the starting reactants, placed in a quartz crucible, was ignited by ohmic heating and was completed within a fraction of a second. After the reaction, the reactor was filled with a large amount of a dark, sponge-like product (characteristic for fibrous material), which was collected for the following analyses.

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